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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:
Pavel LAZAREV and Michael PAUKSHTO

Serial No.: 10/043,959

Filed: January 8, 2002

For: *Device and Method for Forming
Anisotropic Films*

PETITION FOR RETROACTIVE
LICENSE UNDER 37 CFR 5.25

Examiner: Quinn, Mark
Group Art Unit: 2871

Date: December 10, 2002

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Attached hereto is a copy of the following patent applications:

Russian Federation Patent Application Serial No. 2001100534, filed January 10, 2001;

and

PCT Application Publication No. WO 02/056066, filed January 10, 2002.

3. Identification Of Inventor(s), Title Of Invention

Title: Device and Method for Forming Anisotropic Films

Inventors: Pavel Lazarev, 52 Rinconada Circle, Belmont, California 94002
Michael Paukshto, 735 Fathom Drive, #31, San Mateo, California 94404

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4. Transmitted herewith:

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Declaration of Alla Sakharova (2pgs.).

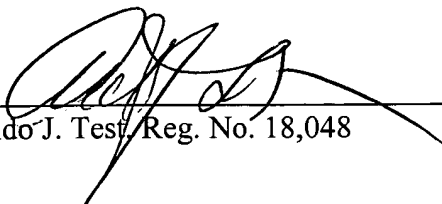
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:
Pavel Lazarev, and Michael Paukshto

**DECLARATION
OF ALLA SAKHAROVA**

For: Patent Application entitled

Device and Method For Forming Anisotropic
Film

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Assistant Commissioner for Patents

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Alla Sakharova declares as follows:

- 10/043 959
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 - (2) The subject matter of the patent application was not under a secrecy order at the time that it was filed abroad, and it not currently under a secrecy order.
 - (3) This license is being diligently sought after she was made aware of the requirement of a license for filing in a foreign country an application for an invention made in the United States.
 - (4) Michael Paukshto was Russian citizen.
 - (5) Pavel Lazarev was a U.S. citizen (former Russian citizen) traveled frequently between the United States and Russia.
 - (6) It is not clear, in fact, where the invention was made by Pavel Lazarev.

- (7) The drafts were prepared and reviewed in Russian only as both inventors were native Russian-speakers.
- (8) The patent was filed abroad because one inventor was Russian citizen and another inventor was traveling frequently between Russia and the United States.
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Title:

Intellectual Property Portfolio Manager
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The method of forming anisotropic films and the device for its implementation

The invention pertains to technology of fabricating thin films, featuring anisotropic properties, such as: optical, magnetic, electric conductivity, etc. In particular, the invention is directed to devices for obtaining polarizing films from liquid-crystalline (LC) solutions of organic materials, for example organic dyes.

Currently, optically anisotropic films obtained from LC solutions of organic dyes are widely utilized especially in fabrication of devices for presenting information [1]. Such films represent thin layers of molecularly ordered organic materials. Flat molecules of the mentioned materials are grouped into orientationally ordered assemblies – supramolecular complexes. The planes of molecules and their dipole moments of optical transition are oriented perpendicular to the axis of macroscopic orientation of the film. In order to create such structure one uses liquid-crystalline state of an organic material solution, in particular – a dye, where molecules already possess local orderliness, while existing in one- or two-dimensional quasi-crystalline aggregates oriented relative to each other. Upon application of such system on the surface of a base and the application of an external orienting action, they assume macroscopic orientation, which in the process of drying not only remains intact, but is also enhanced due to crystallization phenomenon. The polarization axis is directed along the orientation direction coinciding with the direction of application of the polarizer. Peculiarities in the structure of these films necessitate designing special means for their fabrication.

There are various known methods of forming the above-mentioned films and, accordingly, there are various devices for their implementation. See for example [2], where application of an LC solution is performed using a drawing slot or a Mayer's rod; the latter may be of knife-like (doctor-blade), or cylindrical type. Application of an LC solution on the surface of the substrate may be carried out with simultaneous orientation of the supramolecular complexes in a particular direction. The drying process concludes the fabrication process of these films. However, the known devices do not obtain films having reproducible parameters with a high degree of anisotropy. It is believed that this is due to defects in the molecular structure of the film appearing during its fabrication. Besides that, the fabrication process demands long preparation work to develop particular fabrication conditions for each new batch of raw material.

The fabrication of LC films has not been sufficiently studied. Designers face the tasks of enhancing degree of orientation of the resulting films, increasing reproducibility of their parameters, as well as increasing degree of anisotropy of their properties.

The present invention relates to a method and device for fabricating anisotropic films from liquid-crystalline solutions of organic or inorganic materials, in particular optically anisotropic films from LC solutions of organic dyes with high degree of anisotropy and high degree of perfection of their structure (crystallinity), with significant reproducibility of results. The device makes use of a mechanical element

for additional orienting action and creating rheological conditions to increase the degree of perfection of the film's structure.

Applicability of the herein method and the device is not restricted to formation of optically anisotropic films from LC solution of an organic dye. Some films formed from inorganic lyotropic liquid crystals, for example iron oxohydroxide or vanadium oxide feature anisotropic electrical and magnetic properties.

The choice of conditions for fabricating an anisotropic film is determined depending on the utilized materials: their viscosity, volatility of the solvent, ability of the kinetic units in solution to orient and form a structure. In each particular case one can choose corresponding speeds of translation of the mechanical elements in the device and the rate at which to supply solution, as well as the size of the obtained films and distances between elements in the device.

Technical result of the herein invention is the design of the device and the method of fabricating an anisotropic film from LC solutions of organic or inorganic materials, which enhance perfection of the structure of the obtained films, enhance reproducibility of parameters over the surface of the film as well as through its thickness, and also enhance anisotropy of properties.

Technical result of the invention is achieved by the fact that in the device for fabricating anisotropic films from liquid-crystalline solutions, on at least one substrate there is at least one means for supplying solution(s), at least one means of orienting influence on the solution(s) and/or molecules and/or supramolecular complexes, and at least one means of directing the solution implemented in the shape of a plate(s) installed with a possibility to provide solution supply first onto the inner (relative to the orienting element) side of the plate(s) and then onto the substrate(s); moreover, the above mentioned means are implemented with a possibility of translation relative to the substrate holder(s) and with a possibility to fix them at a desired distance from each other and from the substrate holder(s).

In the herein device, one may provide means of forming several films at the same time from various materials, using different substrate holders.

The plate may be implemented as flat, installed at an angle from 0° to 90° to the plane of the substrate holder. At least a part of the plate may be implemented as a cylindrical surface.

In the direction perpendicular to the direction of the relative translation of the above listed means and the substrate holder(s), dimensions of at least one plate may be larger than dimensions of the substrate holder(s), and/or the traverse dimension of the plate edge situated on the side of the substrate holder may be no less than the traverse dimension of the forming film.

At least a part of the plate surface may possess hydrophilic and/or hydrophobic properties. At least a part of the plate surface may feature a relief. Plate(s) may be made out of a polymer material or rubber or at least two various materials, comprising different parts of the plate(s) and/or comprising different layers of the plate(s).

At least one means of the orienting influence may be implemented as knife-like or cylindrical rod, rotating or not. At least one means of orienting influence contains at least one additional plate, one end of which is fixed so that during relative translation of the additional plate and the substrate holder(s) at least a part of the surface of the additional plate translates freely over the substrate surface immediately after the main means of orienting influence.

At least one means of feeding the solution(s) contains at least one injector for supplying solution(s), and/or at least one system of transmitting rollers, and/or at least one channel with a dosimeter of solution(s) supply.

The surface of at least one orienting means may feature a relief.

The device may additionally be equipped with at least one means for removing the solvent from the forming film, which may be implemented as a heating element and/or an air or gas blower unit, and/or a radiating unit, which may be fixed such as to provide processing of at least a part of the substrate surface.

The device may be additionally equipped with a means of providing static conditions, at least one anti-vibration means, and a system of automatic operation and/or fabrication process control.

Technical result of the method of fabrication of an anisotropic film from liquid-crystalline solutions of organic and/or inorganic materials is achieved by the fact that the method includes supplying solution(s) in the way that solution(s) appears on the surface of the solution-directing element and then on the substrate, where it is subjected to an external orienting action; moreover, during fabrication process, the substrate and the means of supplying the solution, the solution-directing element and the means of orienting action are mutually translated in the plane of the forming film.

After and/or in the process of orienting action the forming film is dried.

The element directing liquid crystalline solution is situated at such distance from the substrate and the orienting element, which would provide laminar flow of solution onto the substrate.

The distance between the means of supply, directing and the orienting elements, as well as the distance between the above mentioned elements and the substrate holder are chosen such as to form a film with the desired thickness on the substrate and to provide laminar flow of liquid-crystalline solution upon the contact with the substrate and further flow of solution onto the substrate.

From the supplying element LC solution is fed to the directing element, where preliminary orientation already takes place. Besides that, mechanical elements of the device are installed at such distances from each other and move at such speeds that the flow of solution fed to the substrate becomes laminar. This promotes orientation of kinetic units (elements of the dispersion phase) in solution and obtaining ordered structure throughout the entire thickness of the film. Absence of turbulence during supply and application of the orienting action to the solution decreases defects in the structure induced by disorientation.

The essence of the invention is explained by the following schematics.

Figure 1 is a schematic diagram of a device for fabricating anisotropic films.

Figures 2a and 2b schematically illustrate distribution of the kinetic units in solution (elements of the dispersion phase) during formation of the film: a) in the traditional setup with cylindrical rod, b) according to the disclosed invention, also with cylindrical rod.

The main units of the device, illustrated in the figure 1 are: the substrate holder 1, the means of supplying the dispersion system (liquid crystalline solution of an organic or inorganic material) 2, directing element 3, means of orienting influence 4. The substrate 5, in the process of operation of the device, is installed on the substrate holder 1. The elements 3 and 4 are installed with a possibility of motion relative to the substrate holder 1 such as to provide supply of applying solution over the entire area of the substrate 5 and provide external orienting action on the kinetic units (elements of the dispersion phase) in the solution. Besides that, there is a possibility to install elements 3 and 4 at a desired distance from each other and from the substrate holder. These distances are chosen such as to provide uniform application of solution onto the substrate, formation of laminar flow in the stream of solution applying onto the substrate (Fig.2b), and uniform orienting action on the kinetic units of the solution through the entire thickness of the applying layer. Depending on the viscosity of the solution and the desired thickness of the anisotropic film, in each particular case, one has to determine the mentioned distances and speed of relative motion of elements 3, 4 and the substrate holder. The mentioned operating parameters of the device are determined experimentally or calculated according to the known algorithms.

The means of solution supply is situated and/or implemented such that solution would hit the inner (relative to the orienting element 4) surface of the supplying element 3 and then freely flow onto the substrate under the gravitational force, filling in the space between the elements 3 and 4 over the entire width of the substrate. The edge of the element 3 from which the solution is supplied onto the substrate is usually installed parallel to the longitudinal axis of the rod and, accordingly, perpendicular to the direction of motion.

However, there could be cases when the mentioned edge of the element 3 and the longitudinal axis of the element 4 are not parallel. This would be necessary for obtaining anisotropic films with various characteristics over their area. Various distance between the edge of the element 3 and the element 4 along the longitudinal axis of the element 4 would lead to some non-uniformity of the solution flow onto the substrate and, correspondingly, various orienting action on the kinetic units of the solution in different areas of the substrate.

An example of implementation of the method of fabrication of an optically anisotropic film.

The original material for obtaining optically anisotropic film is aqueous LC solution of sulfonated indanthrone. The dye concentration in the lyotropic LC (LLC) is 7.0 wt. %. Mayer's rod #4 works as the external orienting element, represented by a cylinder wound with a wire, which provides the thickness of the wet layer 9.5 μm . The means of supplying LLC appears as a flat plate, installed at an angle of 60° to the plane of the substrate. The distance between the elements 3 and 4 is 2mm. The speed of movement of

the elements 3 and 4 relative to the substrate is 2.5 mm/sec. After application of LLC, orientating and drying one obtains a film 0.3–0.4 μ m thick, with optical characteristics $K_d = 25$ at $T = 40\%$. i.e. better compared to the traditionally obtained with such systems $K_d = 20-22$ at $T = 40\%$. (K_d - dichroic ratio).

Thus, the suggested method and device for its implementation open new possibilities of obtaining films possessing anisotropic properties (optical, magnetic and other) and perfect structure. Via using the declared invention, one obtains a film with a more perfect structure (and as a consequence, with higher anisotropy of properties) than via analogous methods (in analogous conditions) without using the directing element of the declared design. The presented example does not exhaust all possible uses of the declared invention. One can obtain separate films on different substrate holders, as well as one or several films even with different characteristics on a single substrate holder. The declared device and the method may be used as an independent unit or an operation in any technological line of fabricating anisotropic films. Good results are obtained by using the device with anti-vibration and other facilities, which stabilize application conditions. Besides that, it is recommended to use means of automatic operating and control, especially with computer operation of the process.

Sources of information

1. RU 2155978, 10/09/2000, G 02 B 5/30
2. US 5 739 296. 14/04/1998

Claims

1. Device for fabricating anisotropic films from liquid-crystalline solutions of organic and/or inorganic materials on at least one substrate, containing at least one means of supplying solution(s), at least one means of orienting action on the solution(s) and/or molecules and/or supramolecular complexes, and at least one solution-directing element implemented as a plate(s) installed with a possibility to provide delivery of the solution(s) first to the inner (relative to the orienting element) surface of the plate(s) and then onto the substrate; the above mentioned elements and means are implemented with a possibility of movement relative to the substrate holder(s) and a possibility of their installation at a desired distance from each other and the substrate holder.
2. Device according to the claim 1, wherein the plate is implemented flat and installed at an angle from 0° to 90° to the plane of the substrate holder.
3. Device according to the claim 1, wherein at least a part of the plate is implemented as a cylindrical surface.
4. Device according to any of the claims 1-3, wherein in the direction that is perpendicular to the direction of movement of the above-mentioned elements and means relative to the substrate holder(s), dimensions of at least one plate are larger than dimensions of the substrate holder(s), and/or the size of the plate's edge situated on the side of the substrate holder is no less than the size of the forming film.
5. Device according to any of the claims 1-4, wherein at least a part of the plate(s) possesses hydrophilic or hydrophobic properties.
6. Device according to any of the claims 1-5, wherein at least a part of the plate(s)'s surface features a relief.
7. Device according to any of the claims 1-6, wherein the plate(s) is (are) made out of a polymer material, or the plate(s) is (are) made out of rubber or out of at least two different materials comprising different parts of the plate(s) and/or comprising different layers of the plate(s).
8. Device according to any of the claims 1-7, wherein at least one means of orienting influence is implemented as a knife-like or a cylindrical rod, rotating or not.
9. Device according to any of the claims 1-8, wherein at least one means of orienting influence contains at least one additional plate, one end of which is fixed such that during motion of the additional plate relative to the substrate holder(s) at least a part of the surface of the additional plate is freely translating over the substrate's surface immediately after the main means of orienting action.
10. Device according to any of the claims 1-9, wherein at least one means of supplying solution(s) contains at least one injector for feeding solution(s), and/or at least one system of transmitting rollers, and/or at least one channel with a dosimeter of solution(s) supply.
11. Device according to any of the claims 1-10, wherein on the surface of at least one orienting element there is a relief.

12. Device according to any of the claims 1-11, wherein the device is additionally equipped with at least one means for removing the solvent from the forming film.
13. Device according to the claim 12, wherein the means for removing the solvent from the forming film is implemented as a heating element and/or as an air or gas blower unit, and/or a radiating unit installed such as to provide processing of at least a part of the substrate's surface.
14. Device according to any of the claims 1-13, wherein the device is additionally equipped with a means for providing static conditions.
15. Device according to any of the claims 1-14, wherein it is additionally equipped with at least one anti-vibration means.
16. Device according to any of the claims 1-15, wherein it is additionally equipped with a system of automatic operation and/or control over the fabrication process.
17. Method of fabricating anisotropic film from liquid-crystalline solutions of organic and/or inorganic materials, which includes supplying solution(s) in the way that solution(s) first appear on the surface of the solution-directing element and then on the substrate, where it is (they are) subjected to an external orienting influence; moreover, during film fabrication, the substrate is translated in the plane of the forming film relative to the supplying element, directing element and the orienting element
18. Method according to the claim 17, wherein the forming film is dried after and/or during the orienting action.
19. Method according to the claim 17 or 18, wherein the solution-directing element is placed at such distance from the substrate and the orienting element, which would provide laminar flow of the solution onto the substrate.
20. Method according to any of the claims 17-19, wherein the distance between the means of supply, the directing and the orienting means, as well as the distance from these elements to the substrate holder are chosen such as to obtain a film of a desired thickness on the substrate.

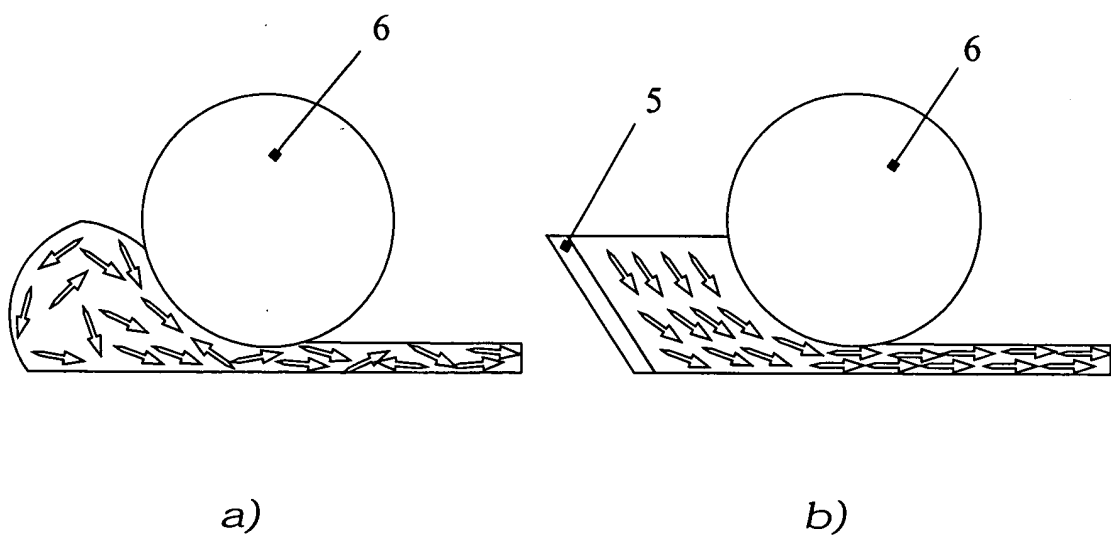
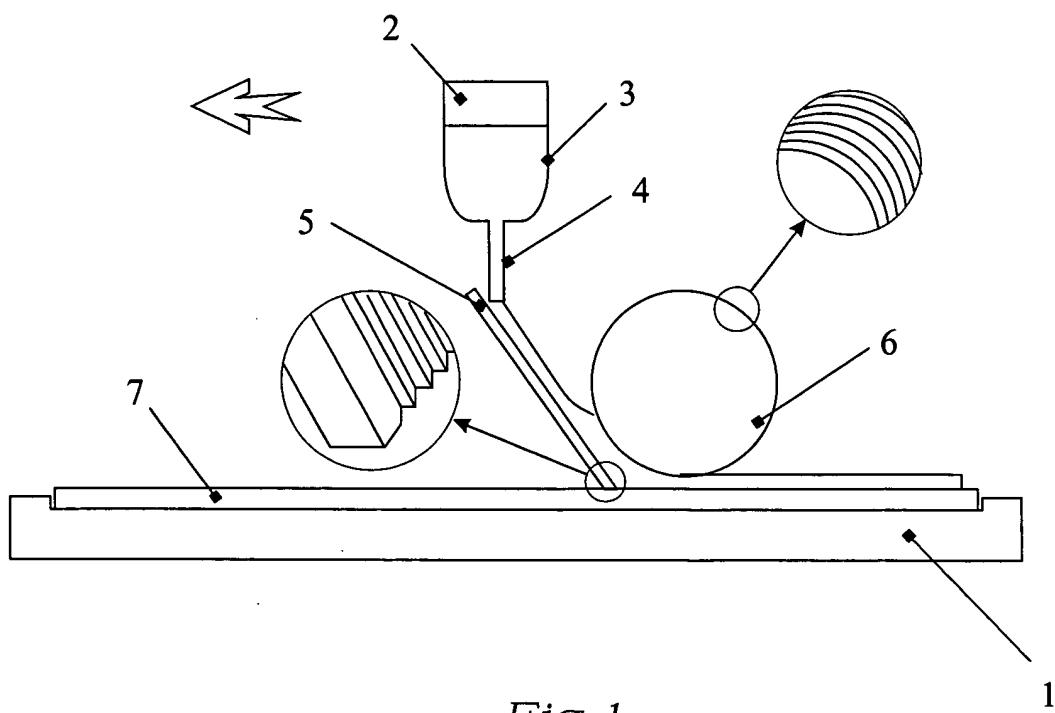


Fig. 2

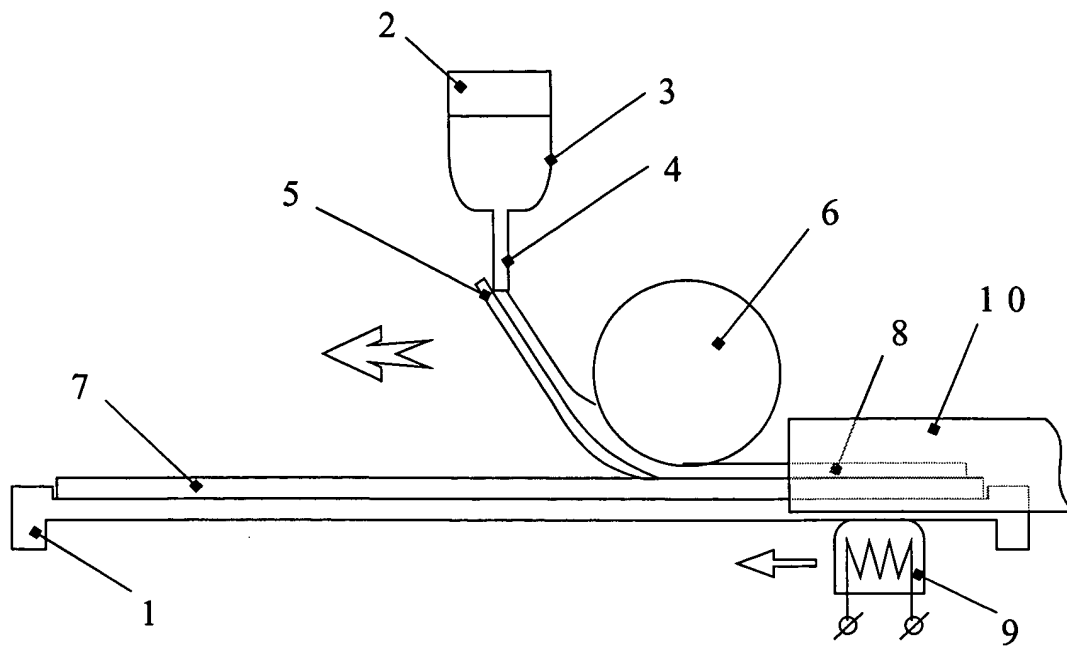


Fig. 3

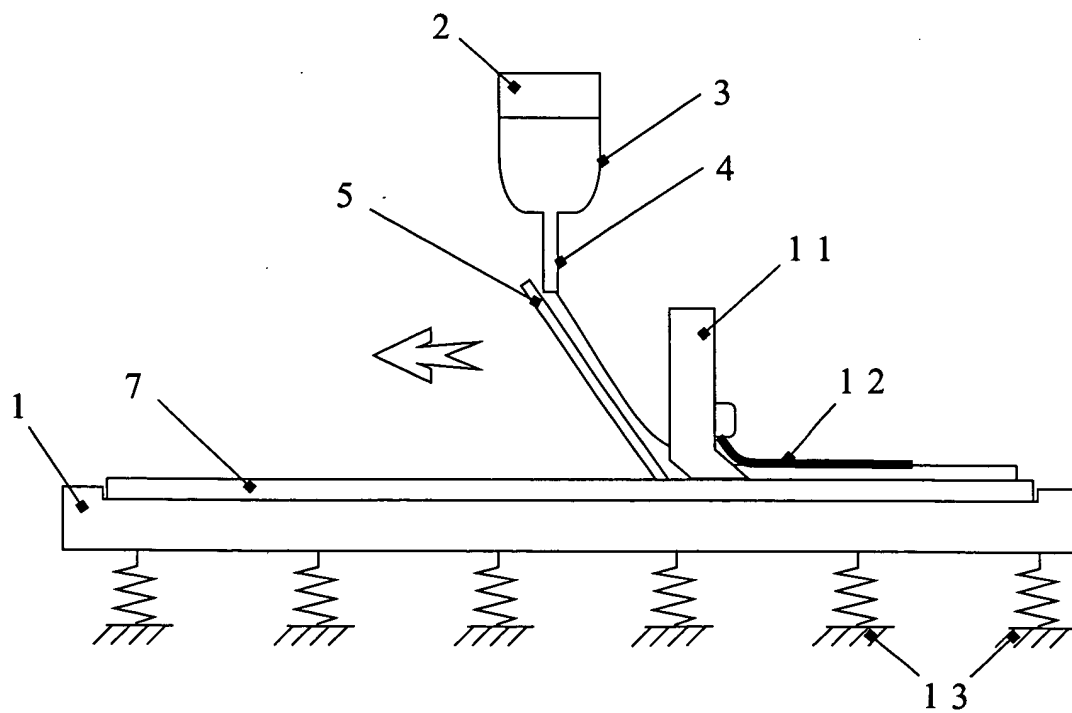


Fig. 4

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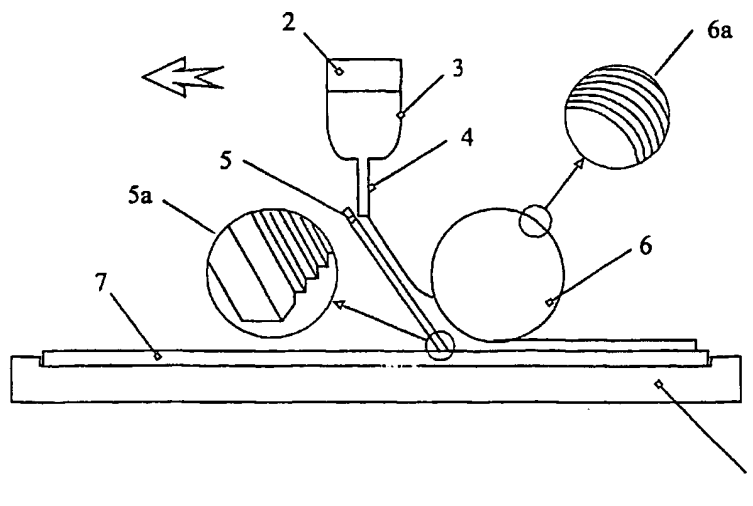
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(54) Title: DEVICE AND METHOD FOR FORMING ANISOTROPIC FILMS



(57) Abstract: This invention pertains to technology of fabricating thin films features anisotropic properties such as: optical, magnetic, electric conductivity and others. In particular, this invention is related to devices for obtaining polarizing films from liquid-crystalline solutions of organic materials, for example organic dyes. The disclosed method and device permits the fabrication of anisotropic films from liquid-crystalline solution with high characteristics and high degree of perfection of the oriented structure with significant reproducibility of results. The device makes use of a mechanical element for receiving and guiding the liquid crystal solution onto a substrate and then providing an orienting action to the kinetic elements as they are delivered.



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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

DEVICE AND METHOD FOR FORMING ANISOTROPIC FILMS

Related Applications

This application claims priority to Russian Application Serial No. RU 2001100534 filed
5 January 10, 2001.

Field of the invention

The invention pertains to technology of fabricating thin films, featuring anisotropic properties, such as: optical, magnetic, electric conductivity, etc. In particular, the invention is directed to devices and methods for obtaining polarizing films from liquid-crystalline (LC)
10 solutions of organic materials, for example organic dyes.

Related Art

Currently, optically anisotropic films obtained from LC solutions of organic dyes are widely utilized especially in fabrication of devices for presenting information. (See RU 2155978, 10/09/2000, G 02 B 5/30.) Such films represent thin layers of molecularly ordered organic
15 materials. Flat molecules of the mentioned materials are grouped into orientationally ordered assemblies – supramolecular complexes. The planes of molecules and their dipole moments of optical transition are oriented perpendicular to the axis of macroscopic orientation of the film. In order to create such structure one uses liquid-crystalline state of an organic material solution, in particular – a dye, where molecules already possess local orderliness, while existing in one- or
20 two-dimensional quasi-crystalline aggregates oriented relative to each other. Upon application of such system on the surface of a base and the application of an external orienting action, they assume macroscopic orientation, which in the process of drying not only remains intact, but is also enhanced due to crystallization phenomenon. The polarization axis is directed along the orientation direction coinciding with the direction of application of the polarizer. Peculiarities in
25 the structure of these films necessitate designing special means for their fabrication.

There are various known methods of forming the above-mentioned films and, accordingly, there are various devices for their implementation. See for example U.S. 5,739,296, where application of an LC solution is performed using a drawing slot or a Mayer's rod; the latter may be of knife-like (doctor blade) or cylindrical type. Application of an LC solution on
30 the surface of the substrate may be carried out with simultaneous orientation of the supramolecular complexes in a particular direction. The drying process concludes the fabrication process of these films. However, the known devices do not obtain films having reproducible parameters with a high degree of anisotropy. It is believed that this is due to

defects in the molecular structure of the film appearing during its fabrication. Besides that, the fabrication process demands long preparation work to develop particular fabrication conditions for each new batch of raw material.

The fabrication of LC films has not been sufficiently studied. Designers face the tasks of enhancing degree of orientation of the resulting films, increasing reproducibility of their parameters, as well as increasing degree of anisotropy of their properties.

Summary of the Invention

The present invention relates to a method and device for fabricating anisotropic films from liquid-crystalline solutions of organic or inorganic materials, in particular optically anisotropic films from LC solutions of organic dyes with high degree of anisotropy and high degree of perfection of their structure (crystallinity), with significant reproducibility of results. The device makes use of a mechanical element for additional orienting action and creating rheological conditions to increase the degree of perfection of the film's structure.

Applicability of the herein method and the device is not restricted to formation of optically anisotropic films from LC solution of an organic dye. Some films formed from inorganic lyotropic liquid crystals, for example iron oxohydroxide or vanadium oxide feature anisotropic electrical and magnetic properties.

The choice of conditions for fabricating an anisotropic film is determined depending on the utilized materials: their viscosity, volatility of the solvent, ability of the kinetic units in solution to orient and form a structure. In each particular case one can choose corresponding speeds of translation of the mechanical elements in the device and the rate at which to supply solution, as well as the size of the obtained films and distances between elements in the device.

Technical result of the herein invention is the design of the device and the method of fabricating an anisotropic film from LC solutions of organic or inorganic materials, which enhance perfection of the structure of the obtained films, enhance reproducibility of parameters over the surface of the film as well as through its thickness, and also enhance anisotropy of properties.

Technical result of the invention is achieved by the fact that in the device for fabricating anisotropic films from liquid-crystalline solutions, on at least one substrate there is at least one means for supplying solution(s), at least one means of orienting influence on the solution(s) and/or molecules and/or supramolecular complexes, and at least one means of directing the solution implemented in the shape of a plate(s) installed with a possibility to provide solution supply first onto the inner (relative to the orienting element) side of the plate(s) and then onto the substrate(s); moreover, the above mentioned means are implemented with a possibility of

translation relative to the substrate holder(s) and with a possibility to fix them at a desired distance from each other and from the substrate holder(s).

In the herein device, one may provide means of forming several films at the same time from various materials, using different substrate holders.

5 The plate may be implemented as flat, installed at an angle from 0° to 90° to the plane of the substrate holder. At least a part of the plate may be implemented as a cylindrical surface.

10 In the direction perpendicular to the direction of the relative translation of the above listed means and the substrate holder(s), dimensions of at least one plate may be larger than dimensions of the substrate holder(s), and/or the traverse dimension of the plate edge situated on the side of the substrate holder may be no less than the traverse dimension of the forming film.

At least a part of the plate surface may possess hydrophilic and/or hydrophobic properties. At least a part of the plate surface may feature a relief. Plate(s) may be made out of a polymer material or rubber or at least two various materials, comprising different parts of the plate(s) and/or comprising different layers of the plate(s).

15 At least one means of the orienting influence may be implemented as knife-like or cylindrical rod, rotating or not. At least one means of orienting influence contains at least one additional plate, one end of which is fixed so that during relative translation of the additional plate and the substrate holder(s) at least a part of the surface of the additional plate translates freely over the substrate surface immediately after the main means of orienting influence.

20 At least one means of feeding the solution(s) contains at least one injector for supplying solution(s), and/or at least one system of transmitting rollers, and/or at least one channel with a dosimeter of solution(s) supply.

The surface of at least one orienting means may feature a relief.

25 The device may additionally be equipped with at least one means for removing the solvent from the forming film, which may be implemented as a heating element and/or an air or gas blower unit, and/or a radiating unit, which may be fixed such as to provide processing of at least a part of the substrate surface.

30 The device may be additionally equipped with a means of providing static conditions, at least one anti-vibration means, and a system of automatic operation and/or fabrication process control.

35 Technical result of the method of fabrication of an anisotropic film from liquid-crystalline solutions of organic and/or inorganic materials is achieved by the fact that the method includes supplying solution(s) in the way that solution(s) appears on the surface of the solution-directing element and then on the substrate, where it is subjected to an external orienting action; moreover, during fabrication process, the substrate and the means of supplying the solution, the

solution-directing element and the means of orienting action are mutually translated in the plane of the forming film.

After and/or in the process of orienting action the forming film is dried.

The element directing liquid crystalline solution is situated at such distance from the substrate and the orienting element, which would provide laminar flow of solution onto the substrate.

The distance between the means of supply, directing and the orienting elements, as well as the distance between the above mentioned elements and the substrate holder are chosen such as to form a film with the desired thickness on the substrate and to provide laminar flow of liquid-crystalline solution upon the contact with the substrate and further flow of solution onto the substrate.

From the supplying element LC solution is fed to the directing element, where preliminary orientation already takes place. Besides that, mechanical elements of the device are installed at such distances from each other and move at such speeds that the flow of solution fed to the substrate becomes laminar. This promotes orientation of kinetic units (elements of the dispersion phase) in solution and obtaining ordered structure throughout the entire thickness of the film. Absence of turbulence during supply and application of the orienting action to the solution decreases defects in the structure induced by disorientation.

Brief description of drawings

The invention will be more clearly understood from the following description when read in conjunction with the drawings in which:

Figure 1 is a schematic diagram of a device for carrying out the present invention.

Figures 2a and 2b schematically illustrate distribution of the kinetic units in solution (elements of the dispersion phase) during formation of the film: a) in the traditional setup with cylindrical rod, b) according to the disclosed invention, also with cylindrical rod.

Figure 3 is a schematic diagram of a device which includes an element for removing the solvent from the forming film, implemented in the form of a heating element.

Figure 4 is a schematic diagram of a device, wherein the function of the element of the aligning influence is performed by a knife-like blade.

Description of Preferred Embodiments

The main parts of the device illustrated in the Figure 1 are: the substrate holder 1; the element for supplying the liquid dispersion system 2 (liquid crystal solution of an organic or inorganic material), implemented as dispenser 3 and channel 4 for delivering the solution;

guiding or solution-directing element 5; the element of aligning influence 6, represented by a cylindrical rod. The element 5 may be provided with aligning grooves 5a to initially align the kinetic elements which are further aligned by the aligning element 6 which may also include aligning grooves 6a. A part of the plate or element 5 may include a cylindrical surface (not shown) adjacent the substrate.

During operation of the device, the substrate 7 is placed on the substrate holder 1. The elements 2 and 5 are mounted with the possibility of translating relative to the substrate holder 1 such as to provide delivery of depositing solution over the entire area of the substrate 7 and provide external aligning influence on the kinetic units (elements of the dispersion phase) of the solution, Figure 2b. Besides that, there is a possibility to mount elements 2 and 5 at the necessary distance from each other and the substrate holder. These distances are chosen such as to provide uniform distribution of the depositing solution over the substrate, to onset laminar flow in the stream of the depositing solution (Figure 2b), and to provide uniform aligning influence on the kinetic units in the depositing solution throughout the entire thickness of the depositing layer.

The mentioned distances and the rate of relative translation of the elements 2, 5 and the substrate holder are determined depending on the viscosity of the solution and the necessary thickness of the forming anisotropic film in each particular case. The mentioned operating parameters of the device are determined experimentally or calculated using known algorithms.

The means of delivering the solution is situated and/or implemented in such a way that the solution is first delivered onto the inner (relative to the aligning element 6) surface of the guiding element 5, and then, under the influence of gravity, free-flows along the guiding element to align the kinetic units and deliver the solution onto the substrate, filling the gap between the elements 5 and 6 over the entire width of the substrate. The edge of the element 5, which delivers solution onto the substrate, is usually positioned parallel to the longitudinal axis of the rod, and, correspondingly, perpendicular to the direction of translation.

However, there may be cases when the mentioned edge of the element 5 and the longitudinal axis of the element 6 are not parallel. This would be necessary to obtain anisotropic films with their characteristics varying over their area. Different distance between the edge of the element 5 and the element 6 along the longitudinal axis of the element 6 will lead to non-uniform flow of the solution onto the substrate and, correspondingly, different aligning influence on the kinetic units in solution in the different regions of the substrate.

Figure 3 illustrates a device, which besides the substrate holder 1, the element for supplying the dispersion system 2, implemented in the form of dispenser 3 and channel 4 for solution delivery, guiding element 5 and aligning element 6, contains the element for removing the solvent from the forming film 8, implemented in the form of a heating element 9. The device

may additionally contain lid 10, which provides static conditions of the drying process (removing the solvent from the forming film). Simultaneous use of elements 9 and 10 allows providing optimum conditions for fabricating anisotropic films and consistently fabricate films with high optical characteristics.

5 Figure 4 illustrates a device, wherein the function of the element of the aligning influence is performed by a knife-like blade 11. The element of the aligning influence also comprises one flexible plate 12, one end of which is fixed in such a way that during relative translation of the additional plate 12 and the substrate holder 1, a part of the surface of the additional plate is freely moving over the surface of the substrate 7 right after the main element of aligning influence 11.

10 The role of this plate is to perform additional aligning influence on the forming film in the process of removing the solvent, which improves its optical characteristics and increases consistency of parameters over the entire surface of the film. Good results may be obtained when using anti-vibrational elements 13 within the device, which stabilize conditions of deposition and fabrication of the film.

15 An example of implementation of the method of fabrication of an optically anisotropic film is described. The original material for obtaining optically anisotropic film is aqueous LC solution of sulfonated indanthrone. The dye concentration in the lyotropic LC (LLC) is 7.0 wt. %. Mayer's rod #4 works as the external orienting element, represented by a cylinder wound with a wire, which provides the thickness of the wet layer 9.5 μm . The means of supplying LLC

20 appears as a flat plate, installed at an angle of 60° to the plane of the substrate. The distance between the elements 3 and 4 is 2mm. The speed of movement of the elements 3 and 4 relative to the substrate is 12.5 mm/sec. After application of LLC, orientating and drying one obtains a film 0.3–0.4 μm thick, with optical characteristics $K_d = 25$ at $T = 40\%$, i.e. better compared to the traditionally obtained with such systems $K_d = 20-22$ at $T = 40\%$. (K_d - dichroic ratio).

25 Thus, the suggested method and device for its implementation open new possibilities of obtaining films possessing anisotropic properties (optical, magnetic and other) and perfect structure. Via using the declared invention, one obtains a film with a more perfect structure (and as a consequence, with higher anisotropy of properties) than via analogous methods (in analogous conditions) without using the directing element of the declared design. The presented

30 example does not exhaust all possible uses of the declared invention. One can obtain separate films on different substrate holders, as well as one or several films even with different characteristics on a single substrate holder. The declared device and the method may be used as an independent unit or an operation in any technological line of fabricating anisotropic films. Good results are obtained by using the device with anti-vibration and other facilities, which

stabilize application conditions. Besides that, it is recommended to use means of automatic operating and control, especially with computer operation of the process.

Claims

1. A device for fabricating anisotropic films from liquid-crystalline solution of organic and/or inorganic materials on at least one substrate, comprising:
5 at least one means of supplying said liquid crystal solution,
a first means providing orienting action to the solution and/or molecules and/or supramolecular complexes of said solution,
at least one solution-directing element implemented as a plate for receiving the solution onto the substrate at said first means for providing orienting action to the solution, and
10 means for providing relative movement between said substrate and said first means and said solution directing element.
2. A device according to the claim 1, wherein the plate is flat and installed at an angle from 0° to 90° to the plane of the substrate holder.
15
3. A device according to the claim 1, wherein at least a part of the plate includes a cylindrical surface.
4. A device according to any of claims 1, 2 or 3, wherein in the direction that is
20 perpendicular to the direction of relative movement the dimensions of the plate are larger than the dimensions of the width of the forming film.
5. A device according to any of claims 1, 2 or 3, wherein at least a part of the plate possesses hydrophilic or hydrophobic properties.
25
6. A device according to any of claims 1, 2 or 3, wherein at least a part of the plate surface features a relief.
7. A device according to any of claims 1, 2 or 3, wherein the plate is made out of a
30 polymer material.
8. A device according to any of claims 1, 2 or 3 wherein the plate is made of rubber.
9. A device according to any of claims 1, 2 or 3 wherein the plate is made of two
35 different materials.

10. A device according to any of claims 1, 2 or 3, wherein the first means providing aligning action comprises a cylindrical rod or roller or doctor blade.

5 11. A device according to claim 10, wherein the first means providing orienting action comprises at least one additional plate, one end of which is fixed such that during motion of the additional plate relative to the substrate holder at least a part of the surface of the additional plate is freely translating over the substrate surface immediately after the main means of orienting action.

10 12. A device according to any of claims 1, 2 or 3, wherein the surface of said first means providing orienting action includes a surface relief.

15 13. A device according to any of claims 1, 2 or 3, wherein the device additionally includes means for removing solvent from the film.

20 14. A device according to the claim 13, wherein the means for removing solvent from the film is implemented as a heating element and/or a radiating unit installed such as to provide processing of at least a part of the substrate surface.

25 15. A devices as in claim 13 wherein the device for removing solvent from the film is implemented as a blower.

30 16. A device as in claim 13 wherein the device for removing solvent from the film is implemented as a radiating unit.

35 17. A device according to any of claims 1, 2 or 3, wherein it is additionally equipped with at least one anti-vibration means.

18. The method of fabricating an anisotropic film on a substrate from a liquid-crystalline solution of organic and/or inorganic materials comprising the steps of:
supplying the liquid crystalline solution on the surface of a solution-directing element,
allowing the solution to flow along the surface of the solution-directing element onto the surface of the substrate,
translating the substrate relative to the solution-directing element, and

subjecting the solution on the substrate to an orienting influence as the substrate is translated.

19. The method of claim 17, wherein the film is dried.

5

20. The method of claim 18 or 19, wherein the solution-directing element is placed at such distance from the substrate and the orienting element such that the solution flows laminarly onto the substrate.

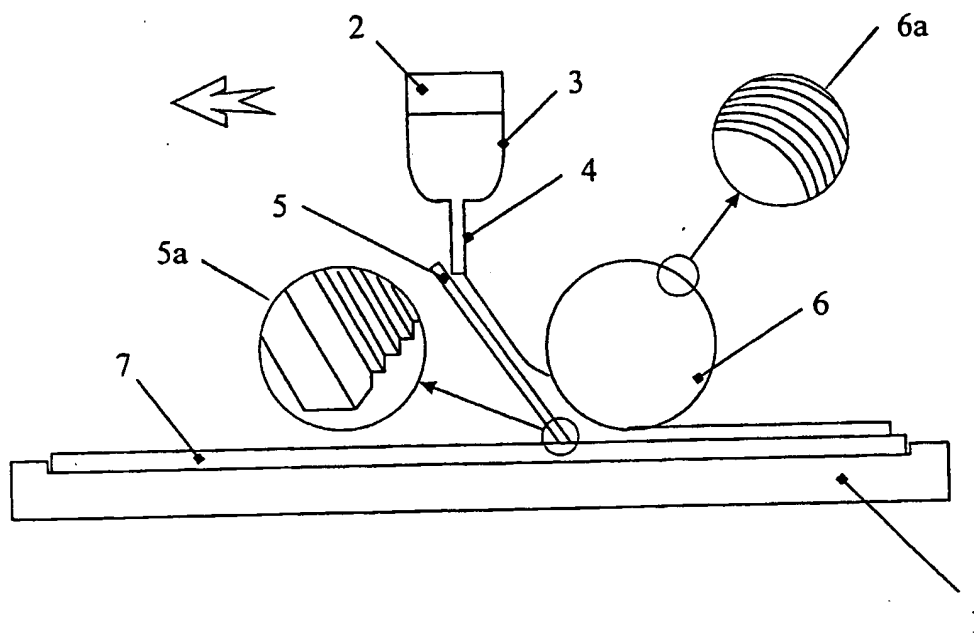


Fig. 1

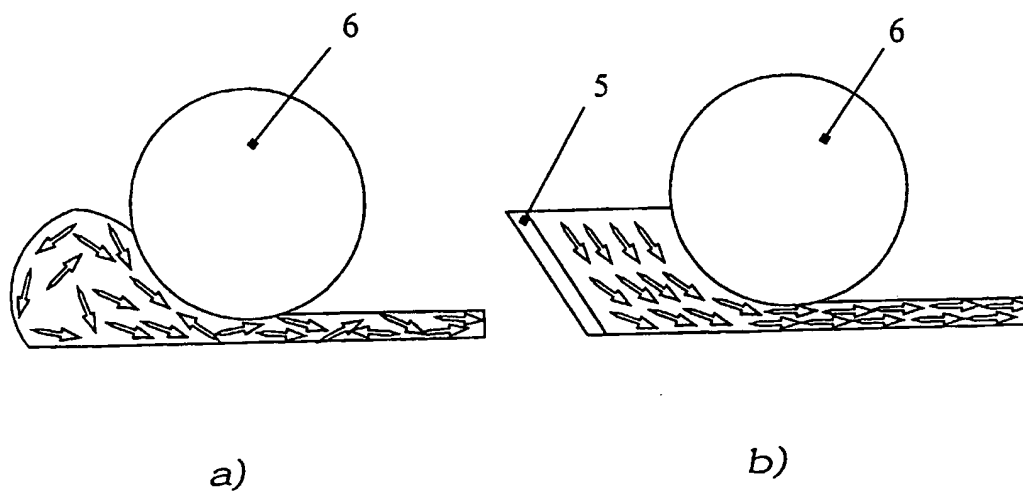


Fig. 2

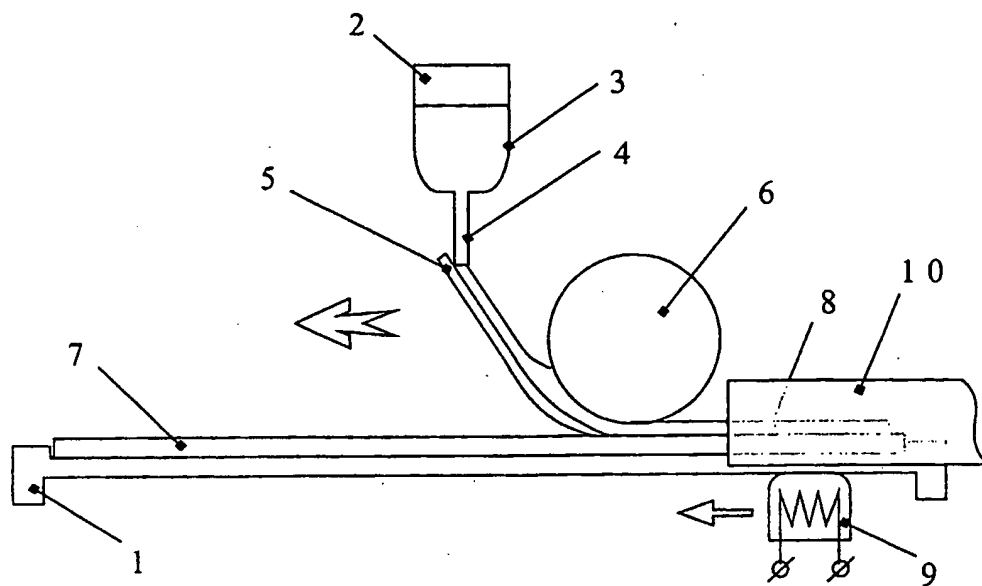


Fig. 3

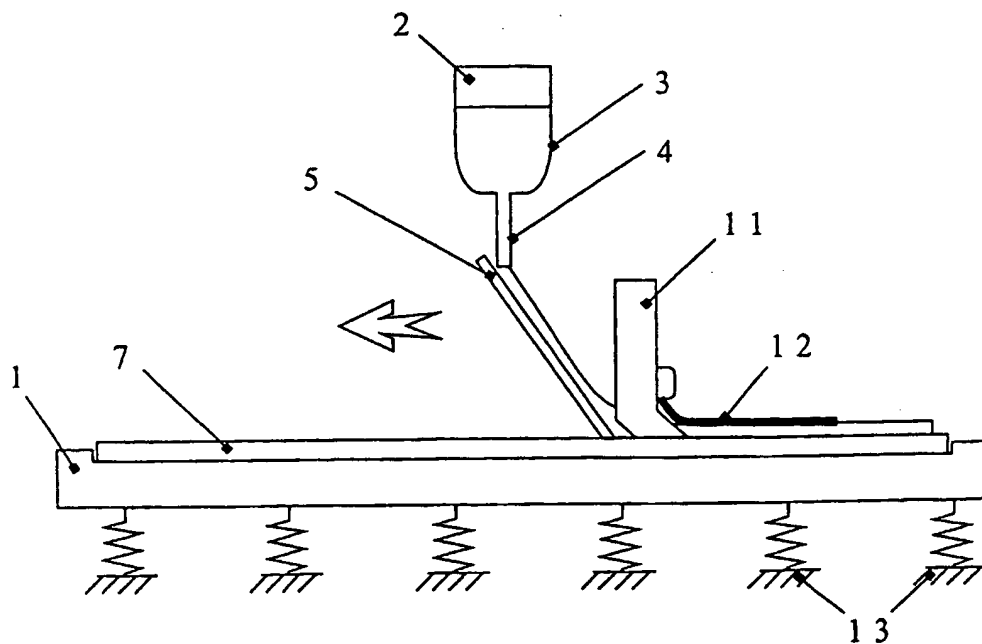


Fig. 4

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 02/00729

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 G02B5/30

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 G02B G02F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 840 145 A (SEIKO EPSON CORP) 6 May 1998 (1998-05-06) page 13, line 13 - line 56; figure 7 -----	1,6,10, 12-16, 18-20
A	US 5 739 296 A (IVANOVA TATIANA D ET AL) 14 April 1998 (1998-04-14) cited in the application column 6, line 35 - line 54 column 8, line 1 - line 52; figures 2,3 -----	1,3,10, 13-16, 18,19
A	US 3 235 631 A (SHELANSKI MORRIS V) 15 February 1966 (1966-02-15) column 1, line 66 -column 3, line 19; figures 1-3 -----	1,10,12, 18,19

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

6 June 2002

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International Application No
PCT/US 02/00729

Patent document cited in search report		Publication date		Patent family member(s)	Publication date
EP 0840145	A	06-05-1998	EP	0840145 A1	06-05-1998
			WO	9743671 A1	20-11-1997
			TW	442692 B	23-06-2001
US 5739296	A	14-04-1998	RU	2047643 C1	10-11-1995
			DE	69428697 D1	22-11-2001
			EP	0700420 A1	13-03-1996
			JP	8511109 T	19-11-1996
			WO	9428073 A1	08-12-1994
			US	6174394 B1	16-01-2001
US 3235631	A	15-02-1966	US	3319519 A	16-05-1967